

PRODUCTION OF Fe-Cr-Ni-Mn ALLOY BY DIRECT SMELTING OF MIXED LOW-GRADE CHROMITE, NICKEL LATERITE AND MANGANESE ORES WITH LOW-GRADE COAL AS REDUCTANT

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ABSTRACT

This study is promoting the concepts of value-added processing and energy conservation by eliminating the energy-intensive processes used in the conventional route of steelmaking through direct smelting of low grade chromite, nickel laterite and manganese ores with low grade coal as reductant. The raw materials, slags and alloy produced were characterized using X-Ray Diffraction Spectrometer, X-Ray Fluorescence Spectrometer, Gravimetric Analysis, Titration method and Atomic Absorption Spectrometry. The metallic alloy produced at 1600°C and three hours of smelting with constant reductant/charged ore of 5% and 8% manganese ore addition was having concentrations of 54.52% Fe, 16.79% Cr, 4.92% Ni, 7.25% Mn and 13.92% Si. The results revealed that the metallic alloy produced from direct smelting method could be a potential substitute raw material for an austenitic stainless steel, particularly in 201 series based on the American Iron and Steel Institute standard.

KEYWORDS: Direct Smelting, Flux, Nickel Laterite Ore, Chromite Ore, Manganese Ore, Reductant & Low-Grade Coal

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INTRODUCTION

Chromium is used in many manufacturing operations particularly, in the manufacture of paints, refractory, leather tanning, wood preservatives, fungicide, dietary supplement, chrome electroplating but most importantly it slows down the chemical reaction that leads to corrosion in stainless steel production. Many minerals contain chromium in low concentrations but the only commercial ore mineral is chromite with a common formula of FeCr_2O_4 depending on the location since the mineral contents of chromite varies from place to place. Chromite is found in peridotite from the Earth's mantle and also occurs in layered ultramafic intrusive rocks and in metamorphic rocks [1]. Chromium is combined with iron and nickel in stainless steel production because of its property as a corrosion inhibitor.

Nickel metal is widely used for many applications including for industrial, military, transport/aerospace, marine and architectural purposes because of its properties such as high resistance to corrosion and oxidation, excellent high-temperature strength and toughness, and relatively low thermal and electrical conductivities [2]. Nickel is used mainly for manufacturing stainless steel. This is the very reason of the increasing demand of nickel for the past years. As far as industrial nickel production is concerned, both hydrometallurgical and pyrometallurgical techniques are currently applied [3]. Dalvi et al.[4] found that nickel occurs in nature principally as sulphides and laterites (oxides and silicates) and the principal source of primary nickel metal at present is

sulphides; however, there is an increasing trend in using nickel laterite ores as the primary source as about 70% of world land based nickel resources are contained in laterites.

Aside from chromium, nickel, and iron, manganese also plays a vital role in steel making. Manganese is an essential alloying element in nearly all types of steel and is used to increase strength, toughness, hardness and hardenability. Manganese is also used for deoxidation and desulphurisation of ferrous metals and alloys [5] and it is also important in the production of cast iron [6]. As an alloying element, it improves the strength, toughness, hardenability, and workability and abrasion resistance of the ferrous products, especially steel [7, 8].

The Philippines is so rich in natural resources especially chromite and laterite ores. According to the U.S Geological Survey [9], the Philippines has laterite reserves of about 1,100,000 metric tons. The biggest deposits of laterite ores are found in Palawan and Surigao and the chromite ore deposits are found in Northern Sierra Madre, Zambales, Samar, Eastern Mindanao, North Central Mindanao, Zamboanga Peninsula and Palawan. However, chromite and manganese reserves in the country are not properly accounted especially in the Zamboanga Peninsula region.

In this study, Fe-Cr-Ni-Mn alloy is extracted from mixed low-grade chromite, nickel laterite and manganese ores by direct smelting method. Direct smelting method is an alternative technology that minimizes the energy requirements in the crude production of stainless steels. It must be noted that the conventional techniques have many disadvantages and one of these is the large energy consumption in recovering the valuable metals from the ores. Apart from the conventional stainless steel process, direct smelting method is a simplified method that aims to produce more value-added products from domestic chromite, nickel laterite and manganese ores.

EXPERIMENTAL PROCEDURE

Materials Used

The chromite ore from Siayan, Zamboanga del Norte, nickel laterite ores from Surigao district and manganese ore from Tungawan, Zamboanga Sibugay in the Philippines were the raw materials used in the study. The chemical composition of the feed materials is shown in Table 1. Lime (CaO) was used as the flux in the direct smelting experiments. The lime was taken from Lazi, Siquijor, Philippines (shown in Table 2). The low-grade coal, used as reductant, was taken from Payao, Zamboanga Sibugay, Philippines (shown in Table 3).

Table 1: Chemical Composition of Feed Materials (wt. %)

Components	Chromite	Saprolite	Limonite	Manganese
Cr ₂ O ₃	40.59	0.54	1.37	-
MgO	13.12	19.78	0.83	1.07
Fe ₂ O ₃	21.71	30.88	84.45	13.42
Al ₂ O ₃	15.95	1.12	2.22	7.72
SiO ₂	4.51	26.75	3.87	13.02
CaO	-	-	-	-
MnO	0.24	0.33	1.53	47.81
Na ₂ O	0.17	-	0.12	0.29
NiO	0.41	2.23	1.87	-
K ₂ O	-	-	-	0.31
P ₂ O ₅	0.04	0.03	0.03	0.04
TiO ₂	0.18	-	0.01	0.91
SO ₃	0.42	0.31	0.47	0.37
LOI	0.30	13.37	1.79	8.84

Table 1: Contd.,				
FC	-	-	-	-
VM	-	-	-	-
Ash	-	-	-	-
S	-	-	-	-

LOI= Loss on Ignition; FC= Fixed Carbon; VM= Volatile Matter

Table 2: Chemical Analysis of Lime

CaO	SiO ₂	MgO	Al ₂ O ₃	Fe ₂ O ₃
55.99	0.2	0.21	0.11	0.05

Table 3: Proximate Analysis of Coal

Fixed Carbon	Volatile Matter	Ash	S
42.64	26.03	31.33	0.21

Preparation of Samples

The limestone, coal, chromite, nickel laterite and manganese ores were crushed and pulverized using mortar and pestle made of aluminum alloy. Prior to the smelting experiments, the ores were calcined using a muffle furnace to remove the excess water, carbonates and some volatile matters. All the test materials passed through a 200-mesh screen using USA Standard Testing Sieve (ASTM E-11). A sample of mixed chromite, nickel laterite and manganese ores with a total weight of 20 grams together with a few percent of coal were mixed together and blended until the mixture became homogeneous. The homogenized mixture was placed in a graphite crucible and compacted with a pressure of 20 kg/cm² using the fabricated die and punch assembly before charging to the elevator furnace.

Equipment Used

All the smelting experiments were conducted in the laboratory elevator furnace which is equipped with a high quality molybdenum disilicide (MoSi₂) heating elements with maximum temperature range of 1,700⁰C. This equipment has electrically driven bottom that is why samples can be loaded easily. The furnace chamber is separated from the heating elements due to the use of a tubular plasma ceramic retort placed vertically in the furnace that also prevents any level of contamination. The equipment was distributed by LABEC Laboratory Equipment PTY. Ltd. Figure 1 presents the schematic diagram of the apparatus.

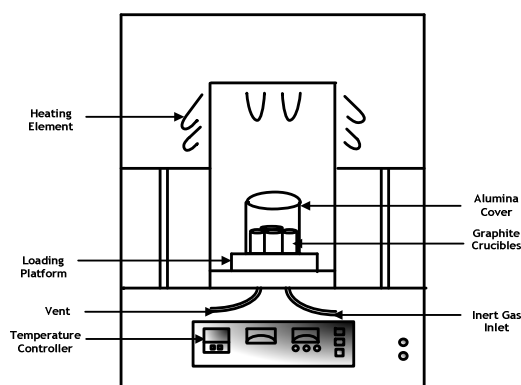


Figure 1: Schematic Diagram of the Elevator Furnace

The smelting experiments were conducted at 1600⁰C in an Argon atmosphere. The mineralogical characterization involved the qualitative identification of phases in chromite and nickel laterite ores by X-ray diffraction (XRD). The XRD

machine used for the analysis of the samples was a Maxima-X Shimadzu 7000 with a CuK α monochromator. The diffractometry was conducted at 40 kV and 30 mA and the data were collected in the range of two-theta values between 3° and 90° with a step size of 0.02° at a speed of 2°/min. XRD patterns were analyzed by the Match! Phase Identification from Powder Diffraction version 3.1.1 Build 58, with reference to the patterns of the Crystallography Open Database [10].

The chemical compositions of the ores were analyzed by X-Ray Fluorescence Spectrometer (XRF). The chemical analysis was carried out using the PANalytical Axios X-ray Fluorescence Spectrometer model PW4400. Quantitative analyses of the Fe-Cr-Ni-Mn alloy and slag phases were also performed using Gravimetric method, Titration method and Atomic Absorption Spectrometry (Shimadzu Atomic Absorption Spectrophotometer model AA-6300).

RESULTS AND DISCUSSIONS

Mineral Composition

The XRD pattern of the chromite ore is shown in Figure 2. The XRD analysis illustrated that spinel ($\text{Mg}_{0.684}\text{Ti}_{0.015}\text{Cr}_{0.833}\text{Mn}_{0.005}\text{Fe}_{0.469}\text{Ni}_{0.003}\text{Al}_{0.988}\text{Si}_{0.003}\text{O}_4$) was the main mineral present in the sample calculated using Match! Phase Identification from Powder Diffraction software. Due to lack of references since the chromite of the study was taken from Siayan, Zamboanga del Norte, the result of the analysis may differ from the reference Crystallography Open Database since the reference was from Southeastern Alps and Outer Dinarides.

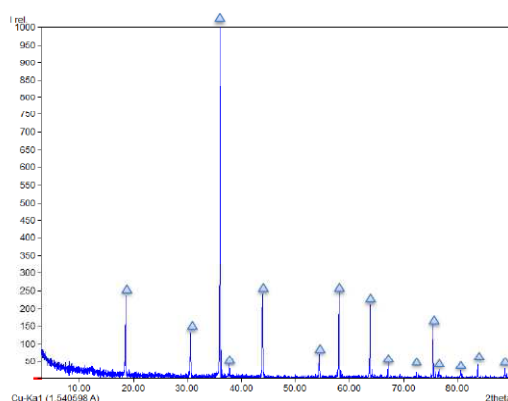


Figure 2: XRD Pattern of Chromite Ore

▲ - Spinel ($\text{Mg}_{0.684}\text{Ti}_{0.015}\text{Cr}_{0.833}\text{Mn}_{0.005}\text{Fe}_{0.469}\text{Ni}_{0.003}\text{Al}_{0.988}\text{Si}_{0.003}\text{O}_4$)

The XRD result of saprolite type nickel laterite ore in Figure 3 revealed that the dominant minerals were the silicate minerals lizardite-1T ($\text{Mg}_3\text{Si}_2\text{H}_6\text{O}_9$) and quartz(SiO_2). The lizardite-1T phase accounted 72.4% and quartz comprised 16.8% of the total phases of the sample. Minor phases were magnetite (Fe_3O_4) with 7% and pentlandite (FeNiS_2) with only 3.9%. There were also unidentified peaks present in the sample. The pattern further indicated that the sample consisted mainly of Mg-silicate mineral in dominant amount which was an indication that the sample was a typical saprolite nickel laterite ore. In saprolite ores, Ni occurs mainly in Mg silicates [11].

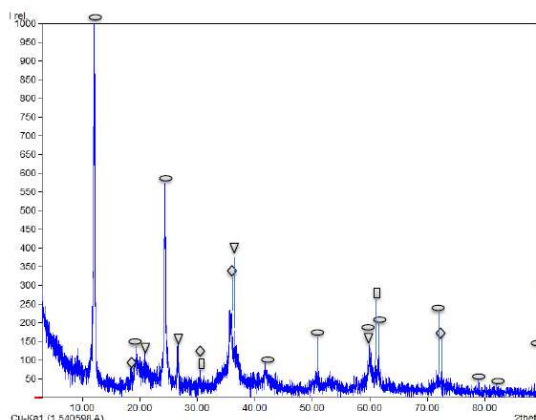


Figure 3: XRD Pattern of Saprolite Type Nickel Laterite Ore

○- Lizardite-1T ($Mg_3Si_2H_6O_9$), ▽- Quartz (SiO_2), ◇- Magnetite (Fe_3O_4), □- Pentlandite ($FeNiS_2$)

Effect of Smelting Time

It was desired to obtain a reputable weight percent of chromium, nickel, manganese and iron in the study. In order to fully understand the reaction mechanism, the homogeneous mixture of ore samples were smelted at different smelting time of 1 hour, 2 hours, 3 hours, and 4 hours. While the smelting time was varied, the reaction temperature and the reductant/charged ore were set constantly at $1600^{\circ}C$ and 5 %, respectively. The reduction of chromite ore with carbon can be represented by the following reaction [12]:



$$\Delta G^0 = 983,772 - 679.6T, J/mol^{-}$$

Under standard conditions, the reduction of chromite ore with carbon will start at temperature above $1175^{\circ}C$. Slag-metal reactions and reduction of metal oxides by gaseous reductants are heterogeneous metallurgical reactions that involve mass transfer between two or more fluid phases. The different phases react in such a way that they follow the reaction steps that take place in succession. The rates of these processes are governed by diffusion theory and the basic laws of chemical kinetics. The former is similar to mass transfer and the latter is temperature-dependent according to the Arrhenius equation.

$$k = A \exp[-Ea / RT] \quad (2)$$

At high temperatures, like in this investigation, the rate constant is larger with smaller activation energy but the rate-controlling step in the present study is not carefully verified since the degree of reduction and the activation energy are not calculated due to the fact that the chemical reactions involved for the three ores used as the test materials of the study are very complicated. It must be noted that in this study, the energy-intensive processes like ferrochromium, ferronickel and ferromanganese in steelmaking productions are omitted and replaced by direct smelting process in which the raw ores of chromite, nickel laterite and manganese are directly smelted to produce the crude alloy.

The reduction of nickel laterite ore with carbon can be illustrated with the reaction



$$\Delta G^0 = 38620 + 35.36T, J/mol$$

Under standard conditions, the reaction of laterite ore with carbon will start at around 820°C. However, the actual smelting temperature will be higher than 1400°C in order to separate the metal from the slag due to their density difference. On the other hand, the reduction of manganese ore follows two stages when reduced at 1300°C. The first stage of reduction involved reduction of the higher manganese and iron oxides to their lower states and the second stage started with random nucleation of iron metal from wustite, both in the interior of the particles as well as on the surfaces exposed to the reducing agent [13]. Then the reduction of MnO and SiO₂ may be represented by the reactions



$$\Delta G^0 = 274470.4 - 162.09T \text{ J/mol}$$



$$\Delta G^0 = 678310.08 - 347.27T \text{ J/mol}$$

The reductions of MnO and SiO₂ under standard conditions start at 1420°C and 1680°C, respectively.

The effect of smelting time on the metallization of iron, chromium, nickel, manganese and silicon as shown in the plot reveals that in the first hour of smelting the concentrations of the metals (Fe, Cr, Ni and Mn) were higher. At the second hour of smelting, the metal concentrations of Fe and Si increased while the metal concentrations of Cr, Ni and Mn decreased. The metal concentrations in the alloy of Fe, Cr, Ni and Si increased after 3 hours of smelting while Mn dropped a little bit. The plot also shows that the Fe concentration was an antithesis of the Cr concentration until the fourth hour of smelting. In the work of McCullough et al. [14], they reported that the concentrations of Cr₂O₃ in the resulting slags were consistently below 0.5% and Cr recoveries were in excess of 95% when chromite ores of Kazakhstan were smelted at 1750°C in 30 minutes using graphite crucibles. They added that the low Cr₂O₃ concentration of the slag was undoubtedly a result of the use of graphite crucibles which resulted in over-reduction and to some extent, the carbon values may be excessive and it was possible that partial contamination of the alloy from the crucible material had occurred. In this work at 1600°C of smelting for 3 hours, there was a complete separation of metal and slag. The lowest concentration of Cr₂O₃ reached 0.8% while the average Cr recovery was 92%.

It can be concluded that the concentration of Fe in the metal decreased beyond 3 hours of smelting while Cr metallization increased due to the fact that iron oxide is less stable than chromium oxide. However, iron oxide was not completely reduced even after 4 hours of smelting because of the iron oxide present in laterite ore and manganese ore. The diluting effect of iron present in the unreduced ores probably affected the nickel recovery in the formed alloy. It can be noticed also from the figure that the metallization of silicon increased gradually up to the fourth hour of smelting. Cardakli et al. [8] noted that the reduction of SiO₂ was not complete and would continue after the 5 h period. The reduction of SiO₂ was a slow process just like MnO and would continue after 5 h of smelting.

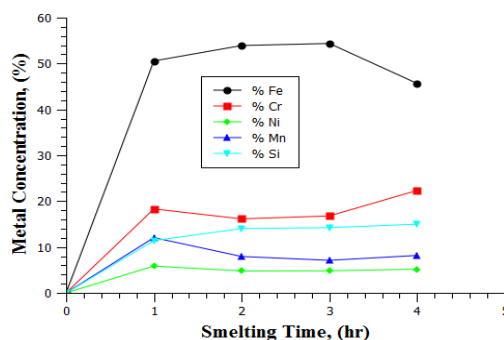


Figure 4: Metal Concentration of the Alloy with Increasing Smelting Time at Constant Reductant/Charged ore (5%) and Temperature of 1600°C

Effect of Ore Composition

A charge mixture containing limonite ore obtained higher Fe content but low in Ni and Cr contents in the metal phase due to the fact that the ore composition of the limonite ore has a high Fe content in the form of Ferric Oxide (Fe_2O_3) containing 84.45% but low in Ni and Cr in the form of NiO (1.87%) and Cr_2O_3 (1.37%), respectively (Table 1). In the case of a charge mixture with saprolite ore containing 30.88% Fe_2O_3 , the concentration of Fe in the alloy was lower compared to the limonite ore with 84.45% Fe_2O_3 , however it contained higher Cr and Ni concentrations of 17.71% and 5.16% in the alloy, respectively. Thus, 15% of Chromite Ore of Zamboanga (COZ) and 85% of Saprolite Ore of Surigao (N2) blend was chosen as the optimum blend of the study.

Figure 5 reveals that as the percentage by weight of saprolite type nickel laterite ore was increased the Cr metal concentration in the alloy decreased while the Ni, Fe, and Si concentrations in the alloy increased. However, there was almost no change in the concentration of the Mn metal in the alloy. The plot shows further that the higher the amount of chromite ore in the charge the higher the Cr concentration in the alloy. It can be deduced that the concentrations of these metals in the alloy were dependent on the type of ores and blend of the charge. Therefore, the optimum charge combination chosen in the study was 85% of saprolite type nickel laterite ore (N2) and 15% chromite ore (COZ) with metallic alloy which was produced in three hours of smelting with 5% coal addition as 56.22% Fe, 17.71% Cr, 5.16% Ni, 0.27% Mn and 10.84% Si.

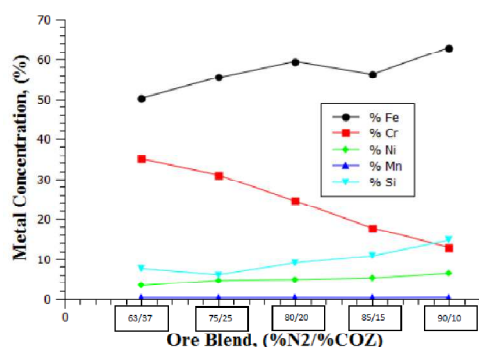


Figure 5: Metal Concentration of the Alloy with Varied Ore Blend at Constant Reductant/Charged Ore (5%) for 3 hrs Smelting Time at 1600°C (N2- Saprolite Ore; COZ- Chromite ore of Zamboanga)

Effect of Flux (CaO) Addition

The data in Figure 6 reveal that lime (CaO) addition increased all the concentrations of the metals (Fe, Cr, Ni, Mn and Si) present in the formed alloy after 2% of CaO/charged ore was added. However, Mn and Si contents

decreased after 4% of CaO/charged ore was added. Fe, Ni and Si contents in the alloy decreased while the concentrations of Cr and Mn increased after adding 6% of CaO/charged ore.

The metal concentrations of the alloy obtained after adding 8% CaO/charged ore were 66.22% Fe, 18.75% Cr, 6.05% Ni, 0.22% Mn and 5.25% Si. This implies further that adding more lime in the system caused the Cr metal concentration to decrease further. Thus, the highest satisfactory level of Ni concentration (6.05%) was achieved. However, the concentration of Ni in the alloy did not satisfactorily reach the standard level of the AISI 304 grade austenitic stainless steel since in the 304 grade the Ni must have a concentration of 8%.

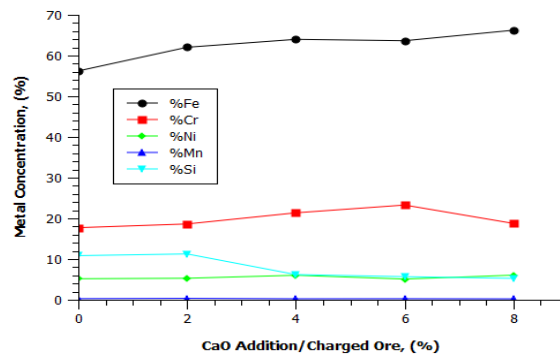


Figure 6: Metal Concentration of the Alloy with Increasing Flux/Charged Ore and 5% Constant Reductant/Charged ore for 3 H Smelting Time at 1600°C

Effect of Manganese ore (MnO) Addition

Addition of manganese ore was studied to obtain an alloy which has a chemical composition that is close to the specified standard from the American Iron and Steel Institute (AISI). The AISI 201 series of austenitic stainless steel contains 16-18% Cr, 3.5-5.5% Ni, 5.5-7.5% Mn, 0.15% C, 0.25% N max and the balance is Fe.

Experiments were conducted by varying the amount of additive (manganese ore) which was added instead of lime. Manganese ore quantity was added as 2%, 4%, 6% and 8% of the charged ore. The results that are reflected in Figure 7 show that adding 8% MnO/charged ore resulted to an acceptable level of Mn concentration which is closer to the specified standard. Increasing the amount of manganese ore in the system is seen to increase the concentrations of Si and Mn in the formed alloy since manganese ore contains more SiO₂ and MnO in the ore, respectively. Thus, manganese ore addition favors the grade of Mn and Si but not to the grade of Fe, Cr and Ni.

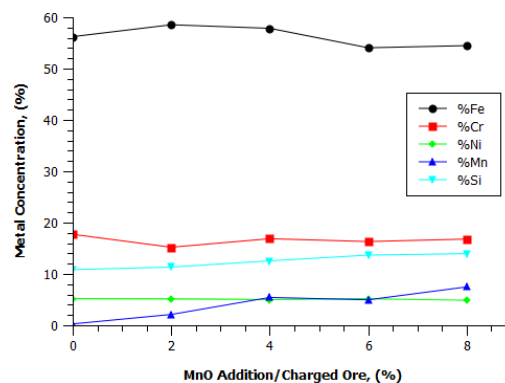


Figure 7: Metal Concentration of the Alloy with Increasing MnO Addition/Charged Ore Ratio and 5% Constant Reductant/Charged Ore for 3 H Smelting Time at 1600°C

Chemical Composition of the Smelted Products

The metallic alloy and its slag obtained at 1600⁰C and three hours of smelting with constant reductant/charged ore of 5% and 8% manganese ore addition per charged ore are shown in Figure 8. It was observed that the metal droplets produced were more spherical in shape compared to the metal droplets produced when lime was added. The weight of the slag decreased significantly while the weight of the metal droplets increased.

The metal droplets contain 54.47% Fe, 16.79%Cr, 4.87%Ni, 7.47% Mn and 13.95% Si The alloy produced from the direct smelting process could be a potential substitute raw material for an austenitic stainless steel, particularly in the typical grade 201 of the American Iron and Steel Institute, in stainless steel manufacturing.



Figure 8: Metallic Alloy and Its Slag Obtained at 1600⁰C and 3 Hours of Smelting with Constant Reductant/Charged Ore of 5% and 8% MnO Addition/Charged Ore

Table 4 presents the comparison of the average experimental analysis and the calculated equilibrium results from Factsage version 7 with registration no. 523. There is a good agreement between the experimental analyses of the metals with that of the Factsage calculated equilibrium results. It can also be seen from the table that the slags still contain higher concentrations of SiO₂ and MgO from experimental and from Factsage calculation. The reason for this is that these oxides are stable so that they were difficult to reduce. Contaminants were also observed in the metal and slag of the experimental results. These are SrO, Cs₂O and Br. The only source of these contaminants was the graphite crucible used in the experiments. It must be noted that the graphite crucibles were made from the waste graphite electrodes from the electric arc furnace provided by a local Ferronickel plant.

Table 4: Comparison of Average Experimental Analysis and Calculated Equilibrium Results from Factsage of Metals and Slags

Average Experimental Analysis Results (wt. %)				Factsage Calculated Equilibrium Results (wt. %)			
Metals		Slags		Metals		Slags	
Mg	0.76	MgO	42.3	Mg	3.88E-03	MgO	41.912
Al	0.45	Al ₂ O ₃	8.82	Al	3.37E-02	Al ₂ O ₃	20.348
Si	13.92	SiO ₂	36.53	Si	12.198	SiO ₂	37.085
P	0.02	P ₂ O ₅	0.007	P	-	Na ₂ O	2.24E-02
S	0.06	SO ₃	0.23	S	-	K ₂ O	1.29E-03
Ti	0.23	TiO ₂	0.14	Ti	0.16212	NaAlO ₂	0.14919
Cr	16.79	Cr ₂ O ₃	1.21	Cr	12.23	KAlO ₂	3.73E-02
Mn	7.25	MnO	2.65	Mn	8.4238	FeO	4.04E-03
Fe	54.52	Fe ₂ O ₃	3.52	Fe	58.417	Fe ₂ O ₃	1.36E-06
Co	0.05	Co ₃ O ₄	-	Co	-	NiO	1.29E-06
Ni	4.92	NiO	0.34	Ni	4.2007	MnO	0.42024
Sr	0.04	SrO	0.12	Sr	-	CrO	1.47E-02

Table 4: Contd.,							
Br	0.86	Br	1.43	Br	-	Cr ₂ O ₃	7.49E-04
		Cs ₂ O	2.27			Ti ₂ O ₃	3.24E-03
						TiO ₂	1.5E-03
						Mn ₂ O ₃	1.67E-05
Weight	4.97 g, ave.	Weight	9.80 g ave.	Weight	7.3214 g	Weight	3.8377 g

CONCLUSIONS

- Direct smelting of mixed low-grade chromite and nickel laterite ores at a temperature of 1600⁰C with a constant reductant/charged ore of 5% for three hours produced an alloy that contains 56.22% Fe, 17.71% Cr, 5.16% Ni, 0.27% Mn, and 10.84% Si without the aid of flux (CaO).
- The highest satisfactory level of Ni concentration (6.05%) was achieved when 8% lime (CaO)/charged ore was added. Results showed that the metal concentrations increased generally except the concentration of Si. It was observed that the flux was able to assist in lowering the slag viscosity as well as help in facilitating a good separation between metal and slag. The alloy produced after the addition of 8% CaO/charged ore contains 66.22% Fe, 18.75% Cr, 6.05% Ni, 0.22% Mn and 5.25% Si.
- Manganese ore addition had a beneficial effect on the metal concentrations of Mn and Si but not on the metal concentrations of Fe, Cr and Ni. The concentration of the metals in the alloy contains 54.47% Fe, 16.79% Cr, 4.87% Ni, 7.47% Mn, and 13.95% Si. The alloy was produced at a temperature of 1600⁰C with a constant reductant/charged ore of 5% and 8% MnO addition/charged ore for three hours of smelting.
- Calculated equilibrium results using Factsage for the metal and slag concentrations were in good agreement with the experimental analysis results.
- Experimental results show that the alloy produced from the direct smelting process using mixed low-grade chromite, saprolite type nickel laterite and manganese ores with low-grade coal as reductant could be a potential substitute raw material for an austenitic stainless steel, particularly in AISI 201series in the production of stainless steel.

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